Union Dyeing Wool/Cotton: Optimizing The Conditions For Polyamino Biguanidine Fixation

Abstract

The efficiency of poly(diethylenetriaminebiguanidine) dihydrochloride, PDETAB, as a pretreatment for union dyeing wool/cotton (62%/38% and 55%/45%) blended fabrics was examined under various conditions of fixative concentration, pretreatment pH, and exposure time. Optimum pretreatment conditions for achieving union shades with good exhaustion, color strength and colorfastness were cold-batching at 20°C with 3% to 4% by weight of fixative at 45 to 60 minutes at pH 6.5. Subsequent to PDETAB pretreatment, dyeing was carried out with wool reactive dyes having alphabromoacrylamido functions.

The quality of union shades for dyeings of 1% to 4% by weight of fabric depended upon the dyebath pH with pH 5.0-6.0 required for 1% and 2% shades and pH 7.0 to 8.0 for 3% and 4% shades. Knit fabrics of an intimate yarn blend of 55%/45% wool/cotton, after PDETAB pretreatment at optimum conditions and dyeing, maintained their original color strengths (KIS) and exhibited no color changes after 25 wash cycles. Not only was PDETAB effective

for achieving union shades on wool/cotton fabric blends; it was equally as effective for union shades when cotton fibers were pretreated before combining them in dyeing with wool fibers that had or had not received the same pretreatment.

Introduction

The prevailing problem associated with the union dyeing of wool and cotton blends from the same dyebath is the preferential uptake of dye by wool. The most efficient dyeing process would involve dyeing wool and cotton with the same dye in one dyebath. We found in past research that the cotton constituent in wool/cotton blends can be selectively pretreated, in the presence of wool, with certain fiber-reactive compounds. This process produced aminized or cationized cellulose that dyed like wool with acid and direct dyes in acidic medium and with wool reactive dyes in nearly neutral dyeing medium.[1,2,3,4] Pretreatment utilized commercial epoxy, azetidinium, and biguanide fiber-reactive compounds produced for dye fixation on cotton. All contained polyamino chains derived from the copolymerization of epoxide, azetidinium, and biguanide compounds with diethylenediamine o diethylenetriamine.

These compounds are used tc improve the physical properties, dyeinç and colorfastness of textiles.[5,6] Polymeric multifunctional epoxides and cationic polyamide azetidinium compounds impart shrink-resistance to wool.[7,8] Similar nitrogen-containing and quaternary ammonium epoxy compounds aminize cotton for property modification and increased dyeability. Specifically, cellulose-epoxide/ epichlorohydrin compounds are used to aminize and cationize cotton for dyeable crosslinked fabrics.[9-14] Azetidinium compounds, formed from the reaction of polyamide with epichlorohydrin, are fundamental to the chemistry of shrink-proofing wool with Hercosett 57 resin, and they were found to increase cotton's dyeability.[15,16,17] Biguanide compounds with reactive amidine functions, have been utilized in the pre- or post-treatments of leather dyeing or printing.[18]

Additionally, the polyhexamethylene biguanide compounds function as bactericides, disinfectants, and antiseptics for skin and cotton textiles.[19,20]

In past research we applied the dye fixatives: Solfix E (Ciba; epoxy), Levogen FSE (Dystar; azetidinium), and Sandene 8425 (Clariant; biguanidine) to wool/cotton textiles from solution by cold batching at 20°C with 20% on the weight of the bath (owb) solutions at pH 11.3. We found that all of the fixatives were effective pretreatments for dyeing wool/cotton blends to union shades with C.I. Acid Red 114. However, overall colorfastness properties of the pretreated and dyed wool/cotton blends were poor-to-moderate but excellent to dryclean-ing and this dictated that they would be serviceable only for outerwear and that they should be refurbished by dryclean-ing.[3] When the epoxy, azetidinium, and biguanide dye fixatives were reexam-

Table I: Union Dyeing of Wool/Cotton Textiles Pretreated with 4% owb PDETAB at pH 9.0 and 70°C: Color Strength and Histogram Analysis.

Dyeing, %*	K/S	Mean Value[a]	Std. Dev.[b]	G-Value[c]
C.L Reactive Blue 69				
no pretreatment, 2% dyeing	-	-	52.69	54.3
1% dyeing	6.78	111	27.65	27.7
2%	11.04	91.2	23.50	23.5
3%	15.43	75.2	20.83	21.1
4%	20.96	62.7	16.72	16.9
C.L Reactive Red 65				
no pretreatment, 2% dyeing	-	-	35.76	33.8
1% dyeing	7.57	130	24.67	24.4
2%	12.09	115	22.89	22.8
3%	17.31	101	20.13	20.3
4%	22.02	92.1	18.48	18.5
C.L Reactive Yellow 39				
no pretreatment, 2% dyeing			17.11	15.2
1% dyeing	7.99	204	14.54	11.9
2%	12.74	205	14.14	11.3
3%	18.41	204	14.45	11.6
4%	21.43	203	14.82	11.7

* Dyed fabrics were pretreated u nless indic 3ted otherwise.
[a] Mean values of the histograms refer to Lhe peak-height positions on the gray-scale axis as measured from the graphs of pixel intensity (y-axis) versus brightness or gray level (x-axis). Higher mean values correspond to lighter shades.
[b] Standard Deviations of the histograms are relative to the range of pixels within the gray-scale range covered by the histogram. Union shades have low values for which the threshold is selected by visual inspection.
[c] G-values correspond directly to standard deviation values. Union shades have low G-values relative to nonunion shades.[21]

Table II: Union Dyeing of Wool/Cotton Textiles Pretreated with 2% owb PDETAB Applied at pH 6.5 and pH 11, 20°C, 30 minutes.

	K/S								
PDETAB Pretreatment pH	Wool/Cotton * % Dyeing			Wool,* % Dyeing			Cotton* % Dyeing		
	1%	2%	3%	1%	2%	3%	1%	2%	3%
C.L Reactive Blue 69									
no PDETAB				12.9	20.4	26.7	0.40	0.45	0.56
with PDETAB									
pH 6.5	6.16	12.8	14.3	11.5	20.2	23.3	3.84	5.49	6.76
pH 11	7.16	12.5	15.3	10.7	20.2	23.1	3.97	5.89	6.27
C.I. Reactive Red 65									
pH 6.5	7.27	13.6	17.6	na	na	na	na	na	na
C.I. Reactive Yellow 39									
pH 6.5	8.90	15.6	18.2	na	na	na	na	na	na

ned under the same application conditions, as pretreatments for union dyeing with C.I. Direct Red 79, C.I. Reactive Red 65, C.I. Reactive Blue 69, and C.I. Reactive Yellow 39, all fixatives were effective but the same colorfastness limitations prevailed except for the biguanide dye fixative. Only the biguanide pretreatment followed by dyeing with C.I. Reactive Red 65, C.I. Reactive Blue 69, and C.I. Reactive Yellow 39 yielded union shades with acceptable colorfastness for laundering.[4] In summary:

- Dyeing with C.I. Acid Red 114, C.I. Direct Red 79, C.I. Reactive Red 65, C.I. Reactive Blue 69, and C.I. Reactive Yellow 39 produced union shades with excellent colorfastness to drycleaning.
- Dyeing with C.I. Acid Red 144 gave poor colorfastness to wet and dry crocking and staining.
- Dyeing C.I. Direct Red 79 gave moderate colorfastness to wet and dry crocking and staining.
- Only the biguanide-pretreated blends that were dyed with C.I. Reactive Red 65, C.I. Reactive Blue 69, and C.I. Reactive Yellow 39, produced the good colorfastness required for home laundering.

Thus, the biguanide fixative, Sandene 8425 liquid, was selected for further study. We report now the optimum conditions for its pretreatment of wool/cotton fabrics to yield union shades when dyed with these wool reactive dyes. Optimum conditions are those that lead to dyeings with high exhaustion, and dyed fabrics with high color strength and colorfastness.

Experimental

Materials:

Fabrics

Wool/cotton union cloth (TF4504), 62% wool/38% pima cotton of 48x44 fabric count and 2/42s worsted wool warp yarns interlaced with 40s/2 combed cotton yarns, TF523, worsted wool flannel, and TF400U, cotton print cloth were obtained from Testfabrics*, Middlesex, NJ. All fabrics were mill scoured and unbleached. Also included in the study was a mill knit fabric composed of intimate blended yarns containing superwash-treated 80s Australian wool and pima cotton (55%/45% wool/cotton) spun on the worsted system by Jagger Brothers (Springvale, Maine) and knit to fabric specifications by Pluma, Inc. (Eden, NC.). In addition to the above-mentioned fabrics, cotton and wool fibers were pretreated individually for dyeing together in the same dyebath. The cotton fibers were obtained in sliver form. Wool fibers were obtained as grease wool from local sheep farmers and it was washed in nonionic detergent.

Pretreatment

The biguanide fixative, Sandene 8425 Liquid, was obtained in hydrochloride form from Clariant Corporation as 55% solids (pH 6.5). The chemical composition of the poly(diethylenetriaminebiguanidine) hydrochloride, PDETAB, $C_4H_{13}N_3 . C_2H_4N_4 . xH_2O_4$ (CAS RN 123502-44-3) is shown in Figure 1.

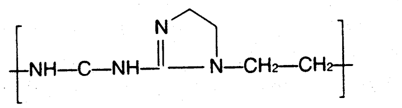

Figure 1- Sandene 8425 liquid is highly cationic, contains over 30% nitrogen, and is highly substantive to cotton. It was designed as a pretreatment to dyeing for chemical modification of cotton with cationic charge.

Dyes

C.I. Reactive Red 65, C.I. Reactive Blue 69, and C.I. Reactive yellow 39 were supplied as Lanasol Red B, Lanasol Blue 3G, and Lanasol Yellow 4G, respectively, by Ciba Corporation.

Method:

Pretreatment Bath:

The PDETAB pretreatment bath was constituted to a 30: 1 liquor ratio (on the weight of the fabric, owf) with 2% to 10% owb Sandene 8425 liquid. The pretreatment bath was applied at pH 6.5 as it was formulated, without pH adjustment. The pH 11.3 bath was constituted from a 2% to 10% owb Sandene 8425 liquid solution by the addition of a 12% sodium hydroxide solution to attain the bath volume and the degree of alkalinity required.

Wetted fabrics were added to PDETAB baths and they were cold-batched for the prescribed reaction times, 1 to 3 hours, at 20°C. Also included in the study were the conditions recommended for the application of Sandene 8425 biguanide fixative from solution: pH 9.0, 4% owb, 15 minutes at 70°C.

After the PDETAB pretreatments, the fabrics were neutralized for three minutes in a 2% acetic acid bath and rinsed thoroughly before dyeing in a fresh bath.

Dyebath:

Dyeings were carried out at pH's within the range of 5.5-6.5 for 1%, pH 6.5-7.5 for 2%, and pH 7.0-8.0 for 3 and 4% dyeings as recommended by the dye manufacturer. A typical time-temperature dyeing profile was as follows: 10 minutes at 20°C followed by temperature rise to 70°C at 1 degree per minute, then dyeing at 70°C for 15 minutes for primary exhaustion, followed by temperature rise to 100°C at 1 degree per minute, and dyeing for secondary exhaustion at 100°C for 30 minutes. After this 150 minute dyeing cycle, the temperature was decreased to 80°C over 15 minutes. Then a clean sodium bicarbonate bath (pH 8.0-8.5) was constituted to aftertreat the freshly dyed fabrics. Dyebath assistants were Albegal B (Ciba), an amphoteric polyglycolether derivative and wool leveling agent, Glauber's salt, and ammonium sulfate.

Testing:

Digital image analysis

Union shade was determined by a digital image analysis system configured with a charged coupled device (CCD) camera linked to a personal computer housing a black/white frame-grabber board and dedicated software operating from a Windows™ environment. The dyed fabrics were processed in black and white digital format. They were displayed as histograms representing pixel distribution within a 255 gray-scale range.

The histograms of union-dyed fabrics showed only one peak (one gray-scale mean value) and this represents the standard for union shade where a union fabric, having wool yarns interlaced by cotton yarns, dyes to one color or grayness value. The unpretreated wool/cotton union fabrics, when dyed with acid, direct, and reactive dyes showed bimodal histograms with two peaks having two gray-scale mean values — one for the lighter cotton yarns that only stained and one for the darker wool yarns that dyed.

The bimodal histograms were analyzed by an in-house curve-fitting program, Abacus, and subsequently an equation was used to arrive at a G-value for goodness of union shade.[21]

Colorimetric analyses

The colorimetric effectiveness of the pretreatment applications was determined by measuring dye-uptake in terms of the fabric's color strength (K/S) from a BYK-Gardner, Inc (Silver Spring, MD) color measurement system that is used to measure the spectral characteristics of dyed fabrics. In the Kubelka-Munk equation, K/S = (1-R)/2R, K/S is directly related to the color intensity of the fabric, where K is the light absorption coefficient, S is the light scattering coefficient, and R is the reflectance factor. The K/S values of the union dyed wool/cotton fabrics were compared to the K/S values for the pretreated wool and cotton after dyeing.

Dyeing Efficiency

Percent dyebath exhaustion was determined by the difference in visible absorption at λ_{max} between the original dyebath and the residual dyebath divided by the absorbance of the original dyebath (x100). These spectrometry readings were made from a Beckman DU-65 Spectrophotometer.

Colorfastness

Colorfastness was measured according to the standard test methods: AATCC Test Method 61—Colorfastness to Laundering, Home and Commercial: Accelerated (Conditions IIA); AATCC Test Method 8—Colorfastness to Crocking, Crockmeter Method (wet and

Table III: Union Dyeing of Wool/Cotton Textiles Pretreated with 4% owb PDETAB at pH 6.5 and pH 11, 20°C, for 1 to 3 hours.

PDETAB Pretreatment		K/S			Mean			Standard Deviation		
		% Dyeing			% Dyeing			% Dyeing		
pH	time	1%	2%	3%	1%	2%	3%	1%	2%	3%
6.5	1 hr	6.8	13 1	14.7	131	96.3	84.3	27.5	26.7	27.3
6.5	3 hr	6.8	12.9	15.7	133	97.3	82.8	27.1	26.1	27.5
11	1 hr	5.6	10.5	16.1	133	106	85.5	30 5	28.4	24.5

Table IV: Colorfastness Wool/Cotton Fabrics Dyed with 2% C.I. Reactive Blue 69 after Pretreatment with 3% to 6% owb PDETAB, pH 6 5 and pH 11, 20°C, 45 minutes to 1 hour.

PDETAB Pretreatment		Colorfastness	
pH	time	Dry Crocking	Wet Crocking
3% PDETAB			
6.5	45 minutes	4	2-3
11.3	45 minutes	4-S	3
4% PDETAB			
6.5	1 hr	4	3
1.3	1 hr	3-4	2
6% PDETAB			
6.5	1hr	4	2-3
11.33	1hr	4	2-3

dry conditions) where "crocking" refers to color loss from wear or abrasion; AATCC Test Method 132—Colorfastness to Drycleaning; and AATCC Test Method 16E—Colorfastness to Light (20, 40 hour increments).

Results and Discussion

1. Wool/Cotton Union Fabrics

a. Prescribed Pretreatment Conditions: 4% owb, pH9.5, 70°C, 20 minutes

In the 1980's, Sandene 8425 was developed to cationize cotton for level dyeing at nearly neutral or slightly acidic pH with anionic reactive dyes.[22] Product information indicated that Sandene should be applied by exhaust at 3 to 6% owb, liquor ratio 10:1 to 30:1, at neutral pH. The process was begun cold and the temperature was raised to 70°C (158°F) within 15 minutes. Soda ash was added to adjust to pH 9.5 to 10.0. The process ensued for 20 minutes. The bath was cooled, drained and the fabric was rinsed. For fabrics that were difficult to penetrate, neutral conditions were prescribed where the fabric was added at 20°C to 30°C in neutral bath for 5 minutes. Then 4% to 5% Sandene 8425 liquid was added and run for 10 minutes, followed by elevating the temperature to 95°C and processing at this temperature for 20 minutes.[23]

Our results of pretreating wool/cotton at pH 9.0, 70°C similarly with 4% owb Sandene 8425 and dyeing with wool reactive dyes are shown in Table I.

With no pretreatment, 2% dyeing with C.I. Reactive Blue 69 (and for the other two dyes, not shown), the histograms of the dyed fabrics showed bimodal pixel distribution about two gray level mean values where their standard deviations and G-values were relatively large as shown Table I. By contrast, all pretreated and dyed fabrics had relatively smaller standard deviations and G-values. Relatively poor union shade was obtained for wool/cotton fabrics dyed to 1% with C.I. Reactive Blue 69. By contrast, 2%, 3%, and 4% dyeings with C.I. Reactive Blue 69 and 1% to 4% dyeings with C.I.Reactive Red 65 and C.I. Reactive Yellow 39 were good union shades. Note that as the dyeing percentage increased, there was an expected and progressive increase in K/S while the standard deviations and G-values decreased. Thus, for C.I. Reactive Blue 69, union shades were easier to attain at higher depths of shade.

Note also that the 1% to 4% dyeings of wool/cotton fabrics with C.I. Reactive Yellow 39 were good union shades as determined by low standard deviations and G-values. This can be explained by the high intensity of the color and the high tinctorial strength of the dye. These observations were affirmed by visual inspections.

Table V: Colorfastness of Wood/Cotton Intimate Yarn Blended Knit Fabrics Pretreated with 3% owb PDETAB at pH6.5 and PH11 for 45 minutes, 20°C.

PDETAB Pretrcatment pH	Dyebath % owb	Dyebath pH	K/S Before Wash	K/S After 25 Wash Cycles	Colorfastness to ColorChange 25 Wash Cycles
pH 6.5	3	5.5-6.5	20.7	20.7	4-5
pH 11	3	6.0-7.5	19.2	20.8	4-5

b. Modified Pretreatment Conditions: 2% owb PDETAB, pH6.5 and 11, 20°C. 30 minutes

In order to find the optimum conditions for union shade for the full 1% to 4% range of dyeings and to modify processing conditions for greater ease and for materials and energy conservation, various fixative concentrations, pH's, temperatures, and exposure times were investigated. Union shades were obtained after pretreating wool/cotton fabrics with 2% owb PDETAB, at pH 6.5 for 30 minutes, 20°C, followed by dyeing with the same wool reactive dyes, as shown in Table II.

These fabrics were analyzed for colorfastness. Colorfastness ratings, on a scale of 1 to 5, refer to a rating of 1 as poor, and 5 as excellent. All of the pretreated and dyed wool/cotton fabrics exhibited ratings of 5 for colorfastness to washing, drycleaning, and 40 hours light exposure. However, colorfastness to washing and staining showed ratings of 3-4 for fabrics dyed with C.I. Reactive Blue 65. Colorfastness to wet crocking was poorest (ratings 2-3) for all dyed fabrics in Table II.

The following observations were made:

- Cotton's ability to take up the dye

was dramatically improved after PDETAB pretreatment The same amount of dye was taken up at either pH.

- Wool's ability to take up dye was relactively unaffected by PDETAB pretreatment.
- Wool/cotton fabrics were dyed to union shades after PDETAB pretreatment but the color strength values (K/S) were less than that of wool alone (not shown for C.I. Reactive Red 65 and C.I. Reactive Yellow 39).
- PDETAB pretreatments at either pH showed nearly the same K/S values for the pretreated and dyed wool/cotton fabrics (not shown for C.I. Reactive Red 65 and C.I. Reactive Yellow 39).

The results suggested that the concentration of PDETAB and pretreatment exposure times should be increased to determine if these conditions would result in higher K/S values and improved colorfastness properties

c. Modified Pretreatment Conditions: 4% owb PDETAB, pH.5 and 11, 20°C, 1 to 3 hours

At 4% PDETAB concentration, the pH of the pretreatment bath was varied from 6.5 to 11.0 and exposure times were varied from 1 to 3 hours. For the determinations of union shades, histogram mean values and standard deviations were included. The results after dyeing with C.I. Reactive Blue 65 are shown in Table III.

Results indicated that acceptable union shades were achieved for all fabrics except those dyed to 1% and 2% after PDETAB pretreatment at pH 11 for 1 hour when the threshold for the standard deviation was set at 27. 5 where the fabrics appear visually to be union shades. Based upon these results, the study was limited to exposure times of 45 minutes to 1 hour and to a PDETAB concentration range of 3% to 6%, with processing at pH 6.5 and 11.

d. Modified Pretreatment Conditions: 3% to 6% owb PDETAB, DH 6.5 and 11 20°C, 45 minutes to 1 hour.

Union shades were obtained for all dyed fabrics pretreated under the conditions shown in Table IV. All colorfastness ratings of these fabrics pretreated with 3% and 6% PDETAB for 45 minutes and 1 hour at both pH's were 3-5 except for colorfastness to crocking where the results are shown below.

The conditions favoring highest colorfastness to crocking were 3% PDETAB applied at pH 11.3 for 45 minutes. However, these dyed fabrics were not the best union shades. when compared to the dyed fabrics pretreated with 3%, 4% and 6% PDETAB at pH 6.5. The best union shades with the highest colorfastness, as shown in Table IV, were obtained as the result of 4% PDETAB pretreatment applied at 20°C, pH 6. 5, 45 minutes to 1 hour.

Table VI: Union Dyeing of Wool/Cotton Fiber blends of Unpretreated and Pretreated Wool Fibers Combined with Pretreated Cotton Fibers. Pretreatment - 3% owb PDETAB, pH 6.5, 45 minutes, 20°C; Dye - C.I. Reactive Blue 69.

PDETAB Pretreatment	Dyeing %	Dyeing pH	% Dyebath Exhaustion	K/S Woo ' Fibers	K/S Cotton Fihers	L[a] Wool/Cotton	a*[b] Wool/Cotton	b*[c] Wool/Cotton
#1. wool pretrealed	2	6.5-7.5	95.9	21.5	16.8	32.0/30.3	-11.4/-12.9	-25.0/-24.
#2. wool unpretreated	2	6.5-7.5	94.8	19.1	17.4	31.4/31.5	-11.3/-12.5	-24.6/-24.7
#3. wool unpretreated	3	7 0-8.0	81.7	23.2	22.3	30.7/28.6	-13.0/-10.7	-25.8/-23.8
#4. wool unpretreated	4	7.0-8.0	71.7	26.7	18.8	28.1/30.5	-12.0/-10.8	-24.5/-24.2
#5. wool unpretreated	4	5.5-6.5	na	45.3	17.3	18.8/30.9	-4.65/-10.8	-22.8/-23.7
#6. cotton pretreated	4	5.5-6.5	na	none	17.1	-/35.7	-/-12.3	-/-24. 9

[a] Lightness/Darkness Value (L)
[b] Difference in Red/Green where "+" = redder and "-" = greener (a*)
[c] Difference in Yellow/Blue where "+" = yellower and "-"= bluer (b*)

Intimate Yarn Blended Fabrics

In preparation for a mill run to pretreat and dye 150 pounds of knit fabrics prepared from 55%/45% Wool/cotton yarn spun on the worsted system, 10 gram fabric samples were pretreated on a laboratory scale at 20°C with 3% PDETAB at pH 6. 5 and pH 11, for 1 hour and and 3 hours before dyeing with C.I. Reactive Blue 69. To obtain union shades at pH 11, the dyebath was adjusted to pH 6.0-7.0 to favor cotton. The results are shown in Table V.

From Table V, the pH of the pretreatment bath has little effect on color stability. Home laundering can be recommended for these fabrics.

3. Wool and Cotton Pretreated in the Fibrous States

If cotton fibers can be pretreated in the fibrous state, there can be a quicker response to fashion dictates. The question of whether the greater surface area of cotton in the fibrous state would require specifically modified pretreatment conditions was addressed by pretreating cotton fibers in sliver form with the PDETAB fixative at optimum conditions as defined above. Note that the pretreatments of wool and cotton fibers were isolated. Note also that these pretreated fibers were combined in dyeing as a 55%/45% wool/cotton fiber blend.

From Table VI, the following observations were made for union dyeing of pretreated cotton:

- For 2% and 3% dyeings at pH 6.5-7.5, union shades can be attained when dyeing in the presence of wool, whether the wool is pretreated or not pretreated.
- 3% dyeings at the higher dyebath pH 7.0-8.0, resulted in good union shades with acceptable exhaustion level.
- Union shades for 4% dyeings at pH 7.0-8.0 were poor and at pH 5.6-6.0, they were unacceptable. Note that the K/S value and dyebath exhaustion for cotton (dyed in the presence of unpretreated wool) at 4% (K/S = 17.3 at pH S.S-6.5 or K/S = 18.8 at pH 7.0-8.0) is lower than that of cotton dyed at 3% (K/S = 22.3). Possibly there were a fixed number of dye sites on cotton and these were exhausted in dyeings above 3%.

In summary, optimum pH for 2% dyeing was 5.5-6.5 and for 3%, pH 7.0-8.0; 4% dyeings were not as effective nor as efficient as 3% dyeings.

Conclusions

Fabric and fiber blends of wool and

cotton were easily dyed to union shades with wool reactive dyes in the same dyebath when the biguanide compound, PDETAB, was used for pretreatment before dyeing with alphabromoacrylamido dyes. Although pretreatment and dyeing conditions favored wool, the dye uptake of cotton in wool/cotton blends increased dramatically. The optimum conditions for pretreatment were room temperature, cold batching with 3% to 4% PDETAB applied at pH 6.5 for 45 minutes to one hour.

True union shades were produced over the dyeing range of 1% to 3% with progressive increases in color strength. The colorfastness was sufficiently high for the requirement of home laundering. Dyeing effectiveness and efficiency was good in terms K/S and dyebath exhaustion, respectively. True union shades depended upon the dyebath pH, that is, pH 6.0-7.0 is recommended for 2% dyeings and pH 7.0-8.0 for 3% dyeings.

There was evidence of a limit of 3% dye sorption by cotton and this suggests that a limited number of cellulose dyesites can be reached or are available. This observation coupled with the fact that union shades can be obtained at pretreatment pH 6. 5 or 1 1 provided the concentration of the biguanide fixative is kept within 3% to 6%, leads to speculation concerning the chemical reactivities of cellulose and the biguanide in this system.

The attraction of biguanide and cellulose did not appear to be pH-dependent. It can be postulated that cotton cellulose undergoes reaction with the biguanide through its endgroups and that there are a lirnited number of them. Hypothetically, the imine group(s) (-C=N-) of the biguanide react with the aldehydic reducing endgroups of cellulose to folm one or both products: 1. a Schiff's base (imine) from the reaction of the allylic chain amadine, and /or 2. an arnide by reaction of the acyclic amidine by ring opening. This would explain the limit to reactivity of cellulose in this system.

The simple and efficient pretreatment and dyeing system described in this work has proven successful not only for union dyeing wooUcotton blended textiles but also for the selective pretreatment of cotton fibers for subsequent dyeing with superwash wool before wool and cotton are blended for the fabrication of yarn and fabric. The successful implementation of this process will open the new market of trans-seasonal wool blends by providing the consumer with casual and comfortable easy-care apparel. □□□

*Mention of a brand or firm does not constitute endorsement by the U.S. Department of Agriculture over other brands or firm names not mentioned

References

(1) J.M. Cardamone, W.N. Marmer, and E.J. Blanchard, "Resin/Amine Pretreatments for Dyeing WooL/Cotton to Union Shades," Proceedings of the 9th International Wool Textile Research Conference, International Wool Secretariat, Biella, Italy (1995).

(2) J.M. Cardamone, W.N. Marmer, E.J. Blanchard, and A.H. Lambert, "Pretreatment of Wool/Cotton for Union Dyeing: Resins Plus Choline Chloride," Text. Chem. Col. (1996) in press.

(3) J. M. Cardamone, G. Bao, W.N. Marmer, and R. L. Dudley, "Pretreatment of Wool/Cotton for Union Dyeing: Fiber-Reactive Quaternary Ammonium Compounds," Text. Chem. Col. (1996) in press.

(4) J.M. Cardamone, G. Bao, A. Francis,and W.N. Marmer, "Modifying Wool/Cotton for Union Dyeing," in Book of Papers 1996 International Conference & Exhibition, AATCC, Nashville, TN, September 1996. Arner. Assoc. Text. Chem. & Col., Research Triangle Park, NC.

(5) D M. Brown and M.T. Pailthorpe, J. Text. Inst. 3, 349-355 (1988).

(6) P.G. Cookson, et al. JSDC 111, 228-236 (1995).

(7) Y. Muraoka and I. Hiraku, Text. Res. J. 64, 514-518 (1994).

(8) K. R. F. Cockett and D. M. Lewis, JSDC 92, 141-149 (1976)

(9) J. D. Guthrie, Text. Res. J. 17, 625-629 (1947)

(10) L. Loeb and L. Segal, Text. Res. J. 24, 654-658 (1954).

(11) S.E. Ellzey, Jr. and C.H. Mack, Text. Res. J. 32, 1023-1033 (1962).

(12) J. B. McKelvey, R.R. Benerito, and T. L. Ward, Indus. Eng. Chem. 6, 115-120 (1967).

(13) R. J. Harper, Jr. et al., Text. Chem. Col. 30, 25-35 (1988).

(14) R. M. Reinhardt and E.J. Blanchard, Polymer Preprints 31, 586-587 (1990).

(15) D. M. Lewis and I. Seltzer, JSDC 88, 93-100 (1972).

(16) S. M. Burkinshaw, et al. JSDC 106, 307-315 (l990).

(17) X.P. Lei and D.M. Lewis, JSDC 106, 352-356 (1990).

(18) M. Dien and W. Egli, Sandoz Patent, Fed. Rep. Ger. DE 3825755 (890216).

(19) D. A. Evans, G.W. Grimshaw, V. J. C. Purdy, UK Patent, GB2182245 (870513).

(20) J.D. Payne, D.W. Kudner, "A New Durable Antiodor Finish for Cotton Textiles," Book of Papers, 1995 International Conference & Exhibition, Amer. Assoc. Text. Chem. & Col., Research Triangle Park, NC (1995).

(21) J.M. Cardamone, and W.C. Damert, "Image Analysis for Detecting Color Uniformity in Dyed Textiles," in Color Technology in the Textile Industry, 2nd ed., G. Celikiz, ed., Amer. Assoc. Text. Chem. & Col., Research Triangle Park, NC, (1996) in press.

(22) J.M. Taylor, JSDC 104, 325-326 (1988).

(23) Anon. "The Sandene Process," Courtaulds Fibres, Derby, England.